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(71) Applicant (for all designated States except US): FASTRAN ENGINEERING LIMITED [GB/GB]; Molly Millars Lane, Wokingham, Berkshire RG11 2PX (GB).

(72) Inventor; and

(75) Inventor/Applicant (for US only): McAULAY, Thomas, Cooper [GB/GB]; Barrule, 1 Manse Lane, Galashiels, Selkirkshire TD1 3RS (GB).

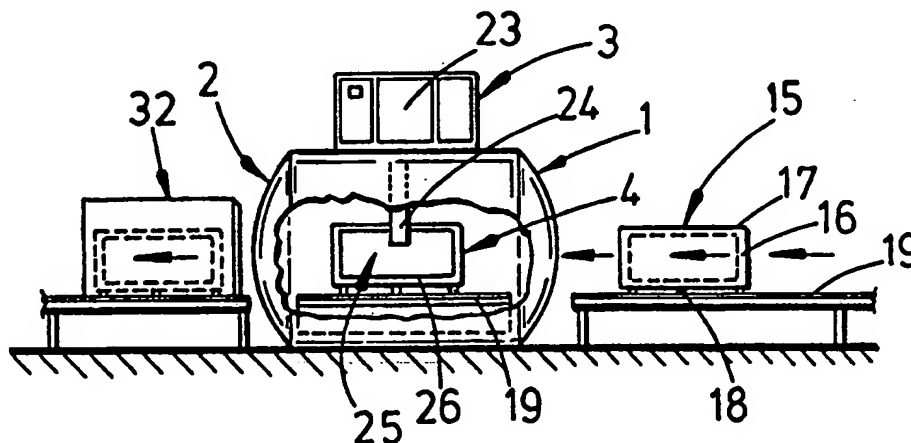
(74) Agents: McCALLUM, William, Potter et al.; Cruikshank &amp; Fairweather, 19 Royal Exchange Square, Glasgow G1 3AE (GB).

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(54) Title: THERMAL TREATMENT



## (57) Abstract

The present invention relates to a thermal treatment apparatus (1) suitable for use in the thermal treatment of a body of matter (5) containing a liquid component. The apparatus (1) comprises a pressure chamber (2) having a support means (4) for supporting the body of matter (5) in an R.F. Heating zone (27) inside the pressure chamber (2) in use of the apparatus (1). Output means such as capacitor plate means (25), of a radio frequency heating device (3), are disposed inside the pressure chamber (2) in proximity to the R.F. Heating zone (27) for heating the body of matter (5) supported in said R.F. Heating zone (27). The pressure chamber (2) has a compressed gas supply means (10) for increasing the ambient pressure inside the pressure chamber above atmospheric pressure, whereby in use of the apparatus (1), the body of matter (5) may be heated up to a temperature above the normal boiling point of the liquid component substantially without vaporisation of the liquid component. The apparatus is particularly suitable for use in the thermal activation of textile treatments.

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THERMAL TREATMENT

The present invention relates to thermal treatment of a wide variety of materials wherein one or more liquids are present which have a boiling point at ambient pressure which is below the optimum or desired treatment temperature. The present invention is especially applicable to thermal fixation treatments of textiles but is also applicable to a greater or lesser extent to cooking and/or sterilisation of food, and to thermal treatment of other materials wherein water is present. Thus this invention provides, inter alia, new and improved methods of thermal treatment and apparatus for use in thermal treatment.

Conventional dyeing and other treatments of textiles generally involve keeping the textile in a boiling solution e.g. dye solution, for several hours. This is highly energy intensive, and results in long processing times with tying down of substantial processing plant infrastructure for long periods of time.

It has previously been proposed to provide special sealed containers for holding the textile and treatment solution, which are then passed through a microwave oven chamber. As the contents of the containers are heated steam is generated increasing pressure inside the containers allowing an elevated temperature above 100°C to be achieved. This system has though some substantial disadvantages. Thus the containers have to be made of special plastics materials which give rise to difficulties in construction of the containers to meet the required pressures and restrict the size of the container that can be used. In addition the formation of steam results in displacement of the dye and uneven dyeing etc.

Similarly thermal treatments of various food materials and products, especially on a large scale, can be relatively time consuming, consume substantial amounts of energy and tie up specialised apparatus for extended  
5 period of time.

It is an object of the present invention to avoid or minimize one or more of the above disadvantages.

The present invention provides a thermal treatment apparatus suitable for use in the thermal treatment of a  
10 body of matter containing a liquid component, which apparatus comprises a pressure chamber provides with a radio frequency heating device, having output means formed and arranged for heating matter within an R.F. heating zone inside said pressure chamber, and a support  
15 means for supporting said body of matter inside said R.F. heating zone, in use of the apparatus; said pressure chamber having a compressed gas supply means for increasing the ambient pressure inside said pressure chamber above atmospheric pressure, whereby in use of  
20 the apparatus, said body of matter may be heated up to a temperature above the normal boiling point of said liquid component substantially without vapourisation of said liquid component.

The present invention also provides a thermal treatment  
25 apparatus suitable for use in the thermal treatment of a body of matter containing a liquid component, which apparatus comprises a pressure chamber having a support means for supporting said body of matter (preferably by means of a holder therefor) in an R.F. Heating zone  
30 inside said pressure chamber, in use of the apparatus; output means such as capacitor plate means, of a radio frequency heating device, said capacitor plates being disposed inside said pressure chamber in proximity to

and at opposite sides of said R.F. Heating zone for heating, in use of the apparatus, of said body of matter supported in said R.F. Heating zone, said pressure chamber having a compressed gas supply means for  
5 increasing the ambient pressure inside said pressure chamber above atmospheric pressure, whereby in use of the apparatus, said body of matter may be heated up to a temperature above the normal boiling point of said liquid component substantially without vapourisation of  
10 said liquid component.

As used herein the expression "body of matter" indicates a body of textile, food or other material which contains at least one liquid component, usually water, which may be bound more or less tightly within the material itself  
15 and/or may be in admixture with said material e.g. in the form of a treatment solution.

The particular form of the R.F. heating device depends on the radio frequencies being used. In general R.F. dielectric heating devices operate in the approximate  
20 range from 1 MHz to 500 MHz and microwave heating devices from 500 MHz to 10 GHz. In the case of R.F. dielectric heating devices the output means are generally in the form of electrodes such as capacitor plates disposed at opposite sides of the heating zone,  
25 or a plurality of rod electrodes disposed in close proximity to one side or at opposite sides of the heating zone.

With a microwave heating device the output means is generally in the form of a waveguide formed and arranged  
30 for supplying energy to the heating zone inside a cavity defined within the pressure chamber. Preferably the waveguide is provided with a microwave window pressure barrier to isolate the elevated pressure within the

pressure chamber from the magnetron, klystron or other microwave energy generator. In order to provide a more even heating of the body of matter, various measures well known in the art such as the use of particular configurations of waveguide and/or stirrer means and the like may be employed.

The present invention is particularly applicable to the thermal fixation of various thermally activated textile treatments, especially dyeing, using aqueous treatment solutions, and allows textile impregnated with an aqueous thermally fixable treatment solutions or suspension to be heated up to a temperature not less than the fixation temperature of said solution or suspension, substantially without formation of steam.

With an apparatus of the present invention dyeing and other treatments of textiles can be effected in just a few minutes or so and thus much more rapidly than with conventional treatments.

It will be understood that the present invention is applicable to all forms of textile including yarn, fibre, filament, tow, top, woven and non-woven or knitted fabrics, etc. including both natural textiles such as wool, cotton, and silk, and synthetic textiles such as polyester, nylon, polypropylene, and acrylic textiles. Furthermore, although the present invention is particularly suitable for dyeing textile material, it is also applicable to various other thermally fixable textile treatments such as bleaching with peroxide and fire resistance treatments using aqueous chemical solutions.

A significant feature of the present invention is that by using a pressure chamber with radio frequency (RF)

heating apparatus output means formed and arranged for heating matter within an R.F. heating zone inside said pressure chamber, the textile being processed does not require to be held within a special RF permeable pressure vessel and a wide variety of convenient holders may be used. Thus, for example, the textile may be held, together with any body of treatment solution, inside a simple pot or box-form vessel, desirably provided with a lid, which may be more or less loose fitting, the vessel and lid being made of an RF permeable material, conveniently a plastics material such as polypropylene or polytetrafluoroethylene (PTFE). The textile could moreover simply be held on a tray and/or conveyor belt, or even supported directly on the floor of the pressure chamber - especially where the textile is in a form which can hold more or less large amounts of fluid, though the latter arrangement would generally be less convenient.

Any suitable compressed gas supply means may be used. Conveniently though there is used a compressor means provided with pressure monitoring means and control means for allowing a desired internal pressure (i.e. ambient pressure within the pressure chamber) to be achieved. Naturally there may be used manually operable control means and/or semi-automatic or automatic control means adapted for providing one or more predetermined pressures.

It will be understood that different treatments and/or textiles may require different temperatures for effective fixation. Thus for example dyeing of polyester may require up to 140°C whilst wool treatment temperatures preferably should not exceed 115°C. In order to prevent steam formation, higher pressures will be required for higher fixation temperatures. Suitable

pressures can be readily determined from reference tables such as the Callendar Steam Tables published by Messrs. Edward Arnold & Co., and/or suitable calculation. In general there would be used a pressure  
5 of from 0 to 100, preferably from 5 to 50 p.s.i. (0 to 7, preferably 0.3 to 3.5 bar or 0 to  $7 \times 10^5$  N/m<sup>2</sup>, preferably  $0.3 \times 10^5$  N/m<sup>2</sup> to  $3.5 \times 10^5$  N/m<sup>2</sup>) above atmospheric pressure, preferably from 10 to 25 p.s.i. (0.6 to 1.75 bar or  $0.6 \times 10^5$  N/m<sup>2</sup> to  $1.75 \times 10^5$  N/m<sup>2</sup>)  
10 above atmospheric pressure.

The temperature of the impregnated textile mass is controlled by adjusting the RF energy output of the RF heating device. The desired RF energy input for any particular treatment temperature may be obtained by  
15 means of calculation. Various formulae may be used for this e.g. in the case of an R.F. dielectric heating device with output means in the form of capacitor plates  
$$P = 69.76 \times W \times SH \times Tr / 60$$
  
wherein P is the required energy in watts/hr, W is  
20 weight of material in Kg, SH is the specific heat of the material (1.0 for water and around 0.3 for textile), and Tr is the desired temperature increase in °C.

Various suitable RF heating devices are known in the art. In general there is used a device tuned to operate  
25 at a radio frequency of from 1 MHz to 10G Hz, preferably with a fundamental frequency of around  $27.12\text{MHz} + 0.6\%$  or low order multiples thereof e.g. 13.65 MHz, 40.68 MHz, 54.2 MHz, and 461.04 MHz for R.F. dielectric heating and 915 MHz and 2450 MHz for  
30 microwave heating, in order to avoid interference with radio signals. The energy output of the RF heating device used in the apparatus of the invention will also depend on the amount of textile to be treated within the chamber at any one time.



The pressure chamber will generally be of non-ferrous material, or at least have a non-ferrous internal liner, in order to avoid induction heating of the chamber. Thus the chamber may conveniently be of e.g. copper,  
5 aluminium, stainless steel or other suitable alloy.

The pressure chamber may be provided with any suitable door means adapted for maintaining a super-ambient pressure within the chamber. Conveniently at least two opposed doors are provided to provide separate entry and  
10 exit points to the chamber to facilitate a through-flow of textile holders.

The present invention also provides a method of thermal treatment of a body of matter containing a liquid component, which method comprises the steps of:  
15 introducing the body of matter into the R.F. Heating zone of an apparatus of the invention;  
increasing the ambient pressure inside the pressure chamber of said apparatus to an elevated pressure above atmospheric pressure;  
20 activating the radiofrequency heating device so as to heat the body of matter up to an elevated temperature not less than an effective treatment temperature thereof, said elevated pressure being at a level not less than that required substantially to prevent boiling  
25 of said liquid component at said elevated temperature;  
deactivating the radio frequency heating device;  
reducing the ambient pressure inside the pressure chamber substantially to atmospheric pressure; and  
removing the treated body of matter from the pressure  
30 chamber.

In a further aspect the present invention provides a method of treatment of a textile using a thermally

- fixable aqueous treatment fluid, which method comprises the steps of:
- impregnating the textile with said treatment fluid;
  - introducing the textile impregnated with treatment fluid
  - 5 into the R.F. Heating zone of an apparatus of the invention;
  - increasing the ambient pressure inside the pressure chamber of said apparatus to an elevated pressure above atmospheric pressure;
  - 10 activating the radiofrequency heating device so as to heat the textile impregnated with treatment fluid up to an elevated temperature not less than an effective treatment temperature thereof, said elevated pressure being at a level not less than that required
  - 15 substantially to prevent formation of steam at said elevated temperature;
  - deactivating the radio frequency heating device;
  - reducing the ambient pressure inside the pressure chamber substantially to atmospheric pressure; and
  - 20 removing the treated textile from the pressure chamber.

The treatment time using an apparatus of the invention will vary with the particular treatment fluid and elevated temperature used, and possibly also the nature and form of the textile being treated. In general

25 though it has been found that effective fixation can be achieved within just a few minutes - as compared with treatment times of several hours in conventional dyeing processes. It will be appreciated that this will give rise to a number of substantial advantages including

30 very substantially reduced energy consumption e.g. a little as 1/40 of the normal requirement in some cases and a major reduction in the substantial infrastructure requirements of conventional processes which include large vessels, machinery to handle these, and high

35 output heating systems, as well as the buildings to

accommodate these. In addition the short treatment times result in substantially reduced degradation of the textile - especially in the case of natural materials such as wool and silk, which in turn results in improved quality end products.

In general treatment times of as little as 3 to 10 minutes may be employed. It will be understood moreover that the textile may be irradiated at the elevated pressure for the full length of time required to achieve fixation or, if desired, for a shorter time with fixation being completed while the treatment fluid is still held inside the pressure chamber at the elevated pressure for a further period of time. In this connection there may conveniently be used an insulated holder for the textile to help reduce the rate of cooling of the treatment fluid after RF R.F. Heating has been stopped and maintain a substantially elevated temperature.

Desirably depressurisation of the pressure chamber is conducted in a controlled manner, e.g. over a period of from 5 seconds to 5 minutes. If desired depressurisation may be delayed until the treatment fluid has dropped to a temperature not substantially greater than that at which steam is formed at atmospheric pressure to limit the amount of steam released though in general it has been found that the fixation is so effective in the case of dyeing, that steam formation at this stage does not give rise to any particular problems. Advantageously there is provided a forced exhaust means such as an extractor fan for removing any steam that may be released during depressurisation.

Further preferred features and advantages of the present invention will appear from the following detailed description given by way of example of some preferred embodiments illustrated with reference to the  
5 accompanying drawings in which:

Fig. 1 is a partly cut-away side elevation of a thermal treatment apparatus of the present invention;  
Fig. 2 a partly cut-away plan view of the apparatus of Fig. 1 with the entrance door open;  
10 Fig. 3 is a partly cut-away end view of the apparatus of Figs. 1 and 2;  
Fig. 4 is a schematic circuit diagram of the RF heating device used in the apparatus of Figs. 1 to 3; and  
Fig. 5 is a view similar to that of Fig. 3 of a second  
15 treatment apparatus of the invention.

Fig. 1 shows a thermal fixation apparatus 1 comprising a pressure chamber 2 provided with a RF heating device 3 and textile support means 4 for holding textile 5 impregnated with a suitable treatment fluid e.g. a  
20 concentrated aqueous dye solution.

In more detail, the pressure chamber is a generally cylindrical stainless steel chamber 2 with entrance and exit doors 6, 7 at opposite ends 8, 9 thereof. The doors 6, 7 are provided with suitable pressure seals and  
25 closing mechanisms (not shown) in generally known manner so as to maintain an elevated pressure in the chamber as required. The chamber 2 is provided with a compressed gas supply means in the form of a compressor 10 with a pressure regulator 11 for establishing a desired  
30 elevated pressure within the chamber 2 via a supply pipe 12. In addition there is provided an outlet conduit 13 with a pressure release valve means 14 for releasing

pressure within the chamber 2 to restore ambient pressure therein. The chamber 2 may of course be of any convenient size. In the illustrated embodiment though it has a diameter of around 3 metres and a length of about 5 metres.

The textile support means 4 comprises a generally box-shaped vessel 15 with insulated walls 16 and a loosely fitting lid 17 mounted on wheels 18 for running along rails 19 upstream and downstream of the chamber 2 and inside it. In order to allow opening and closing of the chamber 2, removable rail sections 20 are utilised in the vicinity of the doors 6, 7. The rails 19 inside the chamber are mounted on a platform 21 supported on the floor 22 of the chamber 2.

15 The RF heating device 3 comprises an RF generator 23 which supplies a high frequency input via suitable supporting conductor strips 24 to generally rectangular capacitor plates 25 which are dimensioned so as to correspond substantially to the sides 26 of the vessels 15 and disposed at opposite sides of an R.F. Heating zone 27 therebetween, close to the sides 26 of the vessels 15. The RF generator 23 is of generally known type rated at 60Kw output and (as shown in Fig. 4) comprises a mains transformer T providing a 10 KV A.C. output which is rectified by a rectifier R to provide a 10KV D.C. supply to an oscillator valve OV which provides a 27 MHz pulsed supply via a capacitor 28 to a first LC circuit 29 which is inductively coupled 30 to a second LC circuit 31 which includes the capacitor plates 25 inside the chamber 2.

Where it is desired to complete cooling of textile and treatment fluid more gradually e.g. in order to provide a longer fixation time and/or minimize thermal shock in

the textile, the vessel 15 may be held inside an insulated holding chamber 32 immediately downstream of the pressure chamber 2.

It will be appreciated that various modifications may be made to the above described apparatus. Thus for example the chamber may conveniently be provided with a pressure relief valve 33 set to open at a predetermined level e.g. 10 bar, above the maximum normal operating pressure and below the safe maximum working pressure of the chamber 2.

Fig. 5 shows an apparatus generally similar to that of Fig. 3 with like parts corresponding to those in Fig. 3 indicated by like reference numerals. In this case though the R.F. heating device comprises a microwave source such as a magnetron 34 which feeds microwaves into a cavity 35 defined within the pressure chamber 2, via a microwave guide 36. The latter is provided with a microwave window pressure barrier 37 conveniently in the form of a toughened glass window which is substantially transparent to microwaves whilst isolating the magnetron 34 from the elevated pressure inside the pressure chamber 2.

#### Example - Dyeing of Wool Tow

3 Kg of wool tow was impregnated with 3 litres of dye solution by conventional padding i.e. spraying the dye fluid onto the fibre as it is passed through the nip between two pad rollers. Each litre of dye solution contained:

15g of commercially available dyestuff Lanaset Blue 2R from Ciba Geigy  
20g of Albegal (Trade Mark) FFA wetting agent (from Ciba Geigy)

10g of Solvitose (Trade Mark) OSA thickener (from the Avebi UK company)  
and sufficient acetic acid to bring the pH down to 4.5.

5 The impregnated wool was loaded into a vessel 4 which  
was then rolled into a pressure chamber 2 of a 20% scale  
version of the apparatus shown in Figs. 1 to 4. The  
pressure inside the chamber was increased to 20 psi (1.5  
bar or  $1.5 \times 10^5$  N/m<sup>2</sup>) and the RF generator switched on  
to deliver 6.5 Kw for four minutes so as to bring the  
10 treatment fluid up to a temperature of 115°C. The RF  
generator was then switched off and the wool held inside  
the chamber under pressure for a further 4 minutes. The  
pressure release valve 14 was opened so as to relieve  
the excess pressure inside the chamber over a period of  
15 30 seconds.

After washing of the treated wool, the dye colour was  
found to be completely fixed and standard inspection  
failed to detect any obvious impairment of the wool  
fibres.

CLAIMS

1. A thermal treatment apparatus suitable for use in the thermal treatment of a body of matter containing a liquid component, which apparatus comprises a pressure chamber provided with a radio frequency heating device,  
5 having output means formed and arranged for heating matter within an R.F. heating zone inside said pressure chamber, and a support means for supporting said body of matter inside said R.F. heating zone, in use of the apparatus; said pressure chamber having a compressed gas  
10 supply means for increasing the ambient pressure inside said pressure chamber above atmospheric pressure, whereby in use of the apparatus, said body of matter may be heated up to a temperature above the normal boiling point of said liquid component substantially without  
15 vapourisation of said liquid component.
2. A thermal treatment apparatus as claimed in claim 1 wherein said radio frequency heating device comprises an R.F. dielectric heating device having output means in the form of electrodes disposed substantially adjacent  
20 said R.F. heating zone.
3. A thermal treatment apparatus as claimed in claim 2 wherein said electrodes are in the form of capacitor plates disposed at opposite sides of said R.F. heating zone.
- 25 4. A thermal treatment apparatus as claimed in claim 1 wherein said radio frequency heating device comprises a microwave heating device having output means in the form of a waveguide formed and arranged for feeding microwave energy to a cavity defined within said pressure chamber.
- 30 5. A thermal treatment apparatus as claimed in claim 4



wherein said waveguide is provided with a microwave window pressure barrier.

6. A thermal treatment apparatus as claimed in any one of claims 1 to 5 wherein said support means for  
5 supporting said body of matter is in the form of a container provided with a lid.

7. A thermal treatment apparatus as claimed in claim 6 wherein said container is made of an RF permeable material.

10 8. A thermal treatment apparatus as claimed in any one of claims 1 to 7 wherein said compressed gas supply means is in the form of a compressor means provided with pressure monitoring means and control means to achieve a desired internal pressure within said pressure chamber.

15 9. A thermal treatment apparatus as claimed in claim 8 wherein said pressure control means is formed and arranged for providing a desired internal pressure in the range of from  $0.6 \times 10^5 \text{ N/m}^2$  to  $1.75 \times 10^5 \text{ N/m}^2$ .

20 10. A thermal treatment apparatus as claimed in any one of claims 1 to 9 wherein said radio frequency heating device is tuned to operate at a radio frequency of from 1 MHz to 10 GHz.

11. A thermal treatment apparatus as claimed in claim 10 when dependent on claim 2 wherein the R.F. dielectric  
25 heating device is tuned to operate at a radio frequency selected from 13.65 MHz and a low order multiple thereof.

12. A thermal treatment apparatus as claimed in claim 10 when dependent on claim 4 wherein said microwave

heating device is tuned to operate at a radio frequency selected from 915 MHz and 2450 MHz.

13. A method of thermal treatment of a body of matter containing a liquid component, which method comprises the steps of:
- 5 introducing the body of matter into the R.F. Heating zone of an apparatus as claimed in claim 1;
  - increasing the ambient pressure inside the pressure chamber of said apparatus to an elevated pressure above
  - 10 atmospheric pressure;
  - activating the radiofrequency heating device so as to heat the body of matter up to an elevated temperature not less than an effective treatment temperature thereof, said elevated pressure being at a level not
  - 15 less than that required substantially to prevent boiling of said liquid component at said elevated temperature;
  - deactivating the radio frequency heating device;
  - reducing the ambient pressure inside the pressure chamber substantially to atmospheric pressure; and
  - 20 removing the treated body of matter from the pressure chamber.

14. A method of treatment of a textile using a thermally activated aqueous treatment fluid, which method comprises the steps of:
- 25 impregnating the textile with said treatment fluid;
  - introducing the textile impregnated with treatment fluid into the R.F. Heating zone of an apparatus as claimed in claim 1;
  - increasing the ambient pressure inside the pressure
  - 30 chamber of said apparatus to an elevated pressure above atmospheric pressure;
  - activating the radiofrequency heating device so as to heat the textile impregnated with treatment fluid up to an elevated temperature not less than the fixation

temperature thereof, said elevated pressure being at a level not less than that required substantially to prevent formation of steam at said elevated temperature; reducing the ambient pressure inside the pressure chamber substantially to atmospheric pressure; and removing the treated textile from the pressure chamber.

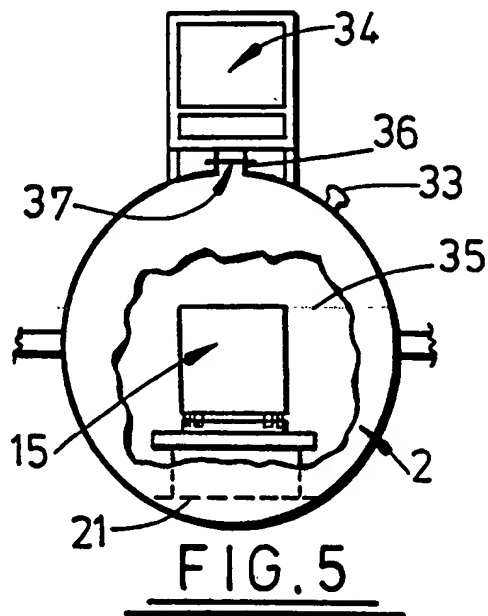
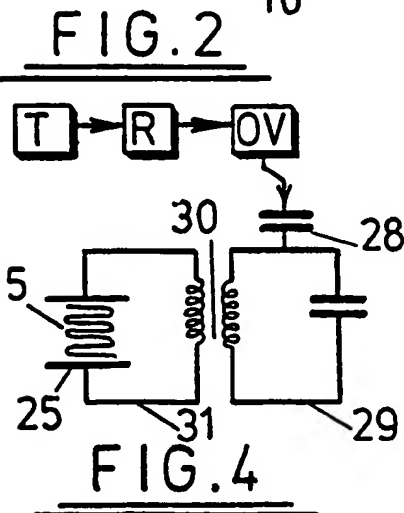
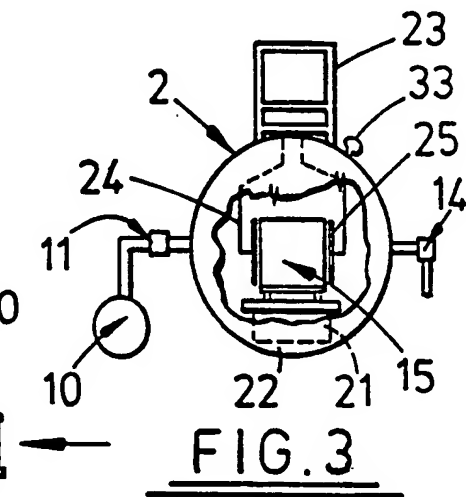
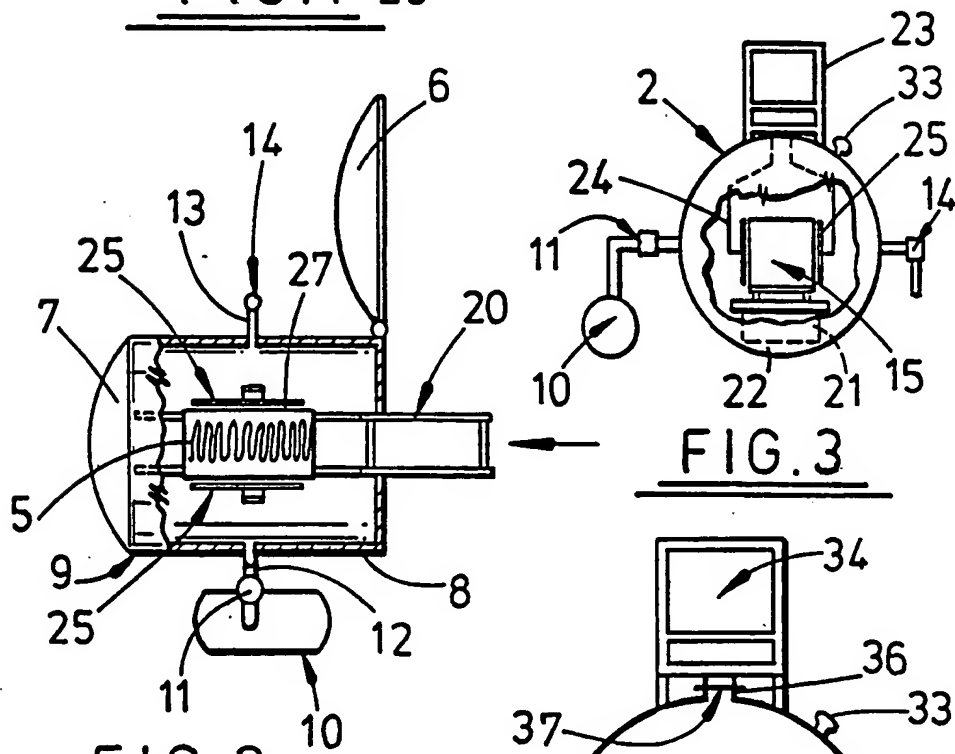
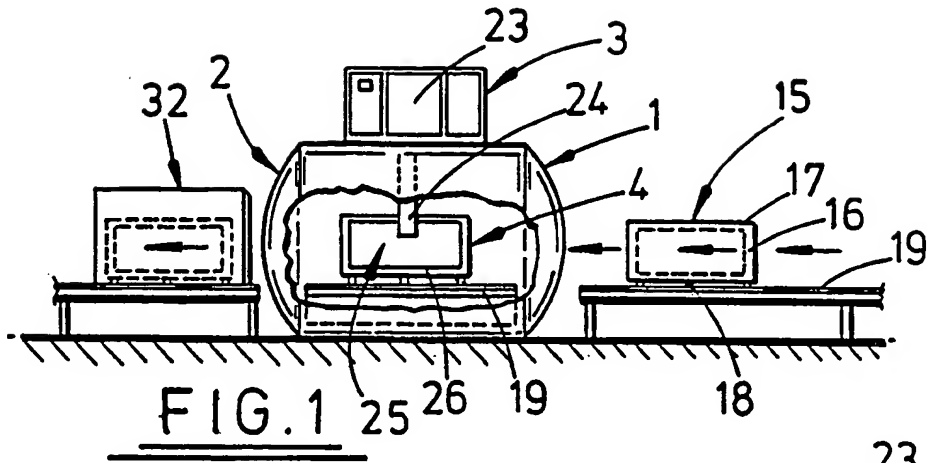
13. A method as claimed in claim 13 or claim 14 wherein said step of reducing the ambient pressure inside the pressure chamber is conducted in a controlled manner over a period of from 5 to 300 seconds.

16. A method as claimed in claim 14 wherein said step of reducing the ambient pressure inside the pressure chamber is delayed until the treatment fluid has dropped to a temperature not substantially greater than that at which steam is formed at atmospheric pressure in order to limit the amount of steam released.

17. A method as claimed in any one of claims 14 to 16 wherein is treated a textile material in the form of at least one of yarn, fibre, filament, tow, top, woven and non-woven or knitted fabric.

18. A method as claimed in claim 17 wherein said textile material is selected from wool, cotton, silk, polyester, polyamide, polypropylene, and acrylic material.

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## A. CLASSIFICATION OF SUBJECT MATTER "

IPC 5 B01J19/12 D06B19/00 H05B6/62 H05B6/80

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 5 B01J D06B D06M H05B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP,A,0 329 338 (ALCAN INTERNATIONAL LTD) 23 August 1989 * abstract * see column 2, paragraph 3 see column 3, line 35 - column 4, line 16 see column 5, line 30 - column 6, line 56 see figures 1-3	1,13,14
A	--- -/--	4,5,8,12

☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP,A,0 016 563 (WOOL DEVELOPMENT INTERNATIONAL LTD) 1 October 1980 * abstract * see page 5, line 9 - line 24 see page 6, line 32 - page 8, line 2 see claim 13 see figures 1,2	1,13,14
A	---	2,3,6,7, 10,11, 17,18
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